NEW TECHNIQUES AND APPLICATIONS OF X-RAY METALLOGRAPHY APPLIED TO WAR PROBLEMS

BY

ROBERT WILSON EYLER

B.S., Monmouth College, 1940

AN ABSTRACT OF A THESIS

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN CHEMISTRY IN THE GRADUATE SCHOOL OF THE UNIVERSITY OF ILLINOIS, 1944

URBANA, ILLINOIS

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PART I

A STUDY OF RECRYSTALLIZATION PHENOMENA IN COLD-ROLLED BRASS

Introduction

Recrystallization occurs in metals which have been subjected to a deforming force and subsequently heat treated. Jeffries and Archer[1] have summarized much of the early experimental work dealing with this subject and have presented theories of recrystallization. These authors suggest that recrystallization begins in fragments of grains produced by deformation. The fragments or nuclei then grow at the expense of strained grains during the process of recrystallization. After the completion of recrystallization, the larger unstrained grains grow at the expense of the smaller unstrained grains.

Doan and Mahla[2] in a later publication suggest two processes occurring during heat treatment—recovery and recrystallization. Recovery is a low temperature process involving loss of internal stress and recovery of original elastic and electrical properties without any change in microstructure. Recrystallization is the formation of new strain-free grains. The number and size of the nuclei for recrystallization are determined by the amount of deformation. These authors state that the amount of deformation is more important in determining the final grain size upon completion of recrystallization than are time and temperature of heat treatment.

This x-ray diffraction investigation of recrystallization phenomena was undertaken in order to provide rather complete information concerning the effects of rather wide ranges of percentage deformation, temperature of heat treatment, and time of heat treatment; to make possible better correlation of x-ray diffraction, microscopic, and hardness data on recrystallization; and to make possible a critical study of the value of the back-reflection diffraction technique.

Experimental Results

X-ray diffraction patterns were made using the back-reflection technique described by Thomas.[3] Patterns prepared from

specimens which had been cold-rolled but not heat treated showed that the effects of deformation were fragmentation of grains, introduction of strain as shown by line-broadening and loss of sharpness of diffraction spots, and the introduction of preferred orientation of grains. The back-reflection diffraction technique was shown to be less satisfactory than a technique involving transmission diffraction patterns in the study of this preferred orientation. Polishing of specimens was found to lead to undesirable surface effects such as surface flow and grain fragmentation. A careful metallographic polishing technique was found to be the most satisfactory method of preparing specimen surfaces. Nitric acid when used as an etch to prepare surfaces of specimens was found to remove strained grains preferentially. Consequently, etching with nitric acid was useful in preparing specimens used for the study of formation of strain-free nuclei but was not satisfactory for general use.

Numerous patterns were prepared from specimens for which the percentage deformation, or the temperature of heat treatment, or the time of heat treatment was varied. These patterns indicated that the amount of deformation was more important than temperature of anneal in the determination of the grain size of the metal at the completion of recrystallization.

A rather extensive study was made of specimens heat treated at 300° C. X-ray diffraction patterns showed quite clearly the formation of strain-free nuclei and the subsequent growth of these nuclei. Hardness and microscopic data were obtained from the same specimens. A comparison of x-ray diffraction and microscopic data indicated that a single grain as seen under the microscope may consist of a number of crystallites as shown by x-ray diffraction.

PART II

DEVELOPMENT OF A RADIOGRAPHIC METHOD FOR LOCATING SMALL DEFECTS IN ALUMINUM ALLOY CASTINGS

Introduction

This work was undertaken at the request of the authorities at Wright Field, Dayton, Ohio. It was desired to develop a radio-

graphic method for the detection of cold-shuts in aluminum alloy castings used in aircraft motors. These defects are due to improper fusion of the metal during the casting process. The defects may lead to failure of the motor during operation, because of the vibrational stresses to which the motor is necessarily subjected. Leakage tests and breaking-up of castings had been rejected as inspection methods because of the uncertainty of the results or the destructive nature of these tests.

Experimental Results

The nature of the defects studied and the low absorption of x-rays by aluminum suggested that any satisfactory radiographic method used must give high radiographic contrast, or in other words, must have high sensitivity in the detection of defects. A comparison was made of the radiographic contrast obtainable from the use of x-ray tubes with tungsten and molybdenum targets. The conventional radiographic tube has a tungsten target, glass windows, and is oil-immersed for cooling purposes. The x-ray tube used in this investigation was water-cooled, and had a molybdenum target and thin beryllium windows. Both tubes were operated at 40 kilovolts tube potential throughout the comparison study. The use of a tube with a molybdenum target was found to lead to higher radiographic contrast, and consequently its use in this study seemed to be justified.

For development of the technique, a casting which had been sawed in such a way as to expose the flaws was used as a test specimen. The parts of the sawed casting were reassembled in their original positions so that a complete casting with known defects in known positions was available for radiographic study. Experimental technique was developed for detecting the cold-shuts using this specimen. Then an unknown casting was submitted as a test of the efficiency of the method. Radiographs were prepared, and then the casting was broken apart and the radiographic results were verified by visual inspection of the indicated faulty regions. This method has been officially accepted by the Army Air Forces Materiel Command at Wright Field.

PART III

OTHER LESS EXTENSIVE INVESTIGATIONS

A Quantitative Study of Stress-Strain Relationships in a Magnesium Alloy

Introduction

This study, undertaken at the request of the War Metallurgy Committee of the National Research Council, represents the preliminary step of a rather extensive research program now being formulated. This program has for its objective the study of the effect of applied stress on the structure of magnesium and other light alloys as a function of geometric shape and as affected by the presence of defects inherent in casting the alloys.

Experimental Results

In this study, test bars were subjected to simple longitudinal stresses using the device described by Clark, Pish, and Seabury.[4] Back-reflection x-ray diffraction patterns were made from the specimen while it was under tension and after release of the tension. The effect of stresses of from 0 to 44,000 p.s.i. was studied. A method involving a nitric acid etch to remove surface layers of the specimen and thus eliminate machining effects was developed.

The x-ray diffraction patterns prepared in this study fixed approximate values for the applied stress necessary to cause shifts in grain positions, grain fragmentation, and broadening of lines on corresponding diffraction patterns. In addition, the study fixed approximate ranges for values of the applied stress necessary to cause elastic changes in the structure of the metal. The results of this study suggested the use of a rotating-film technique to study changes in the diameter of diffraction rings caused by stresses in the elastic range, in future studies involving specimens of varying geometric shape.

Improvement of the Quality of Shell Bodies Made from Alloy Steel

Introduction

This work was undertaken at the request of the Wayne Pump Company, Fort Wayne, Indiana, in order to obtain information

regarding lack of consistency in the performance of artillery shells made from alloy steel. These shells must pass a test involving penetration of armor plate. Certain lots of shells performed satisfactorily in this test, while other lots were unsatisfactory.

Experimental Results

Transmission and back-reflection x-ray diffraction patterns were prepared from thin specimens cut from satisfactory and unsatisfactory lots of shells. These patterns suggested that the heat treatment previously used for strain relief had been ineffective. Heat treatment studies using thin specimens indicated that a temperature of approximately 1000° F. was effective in relieving strain in the metal. The temperature previously used was 375° F. It was suggested that addition agents be used to prevent deterioration of quenching oils used in the manufacturing process; and that age hardening might contribute to more satisfactory performance of shells.

References

1 Jeffries and Archer, "The Science of Metals," McGraw-Hill Book Company, Inc., New York, 1924.

2 Doan and Mahla, "The Principles of Physical Metallurgy," McGraw-Hill Book Company, Inc., New York, 1941.

3 Thomas, J. Sci. Instruments, **18**, 135 (1941).

4 Clark, Pish, and Seabury, J. Applied Phys., **14**, 284 (1943).

VITA

The author was born November 15, 1918, near Monmouth, Illinois, and received his primary and secondary education in the public schools there. After graduation in 1935, he entered Monmouth College and in 1940 received the Bachelor of Science degree, with honors summa cum laude. He was enrolled in the Graduate School of the University of Illinois in 1940 and held a teaching assistantship in the Analytical Division of the Department of Chemistry for four years.

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